REPORT DOCUMENTATION PAGE

AFRL-SR-BL-TR-99-

0105

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REP
	03/31/99	Fina

4. TITLE AND SUBTITLE

Parallel Fabrication and Electronic Characterization of Nanostructured and Nanoheterostructured Metal Surfaces

5. FUNDING NUMBERS

G F49620-96-1-0007

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10. SPONSORING / MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

a. DISTRIBUTION / AVAILABILITY STATEMENT

DISTRIBUTION STATEMENT A
Approved for Public Release
Distribution Unlimited

12. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

Among the more notable accomplishments during the course of this contract we identify the following results: • A precisely ordered and precisely located array of 5 nm diameter nanoclusters has been fabricated by first etching into the substrate an array of holes with diameters comparable to the size of nanoclusters sought and then depositing adatoms on the substrate. Our methods enable rapid fabrication of arrays for fundamental studies and provide a route to manufacturability of nanostructure arrays for technological purposes.• We reported the ability to control the morphology of nanometer thick Ti oxide films which were created via a parallel nanofabrication process using a two-dimensional protein crystal as a template. Atomic force microscopy was used to examine the evolution of these structures from a periodic array of nanometer-scale dots (nanodots) to a screen containing a periodic array of nanometer-scale holes (nanoscreen) as the film thickness was increased. • We reported the creation of large arrays of nanometer-scale dots (nanodot arrays) with the oxides of several additional metals near Ti in the periodic table. • A computer simulation of nanoscale hole formation based on curvature dependent sputtering and surface self-diffusion was formulated. The model simulates the experimental data from our nanopatterning process quite well.

14. SUBJECT TERMS

nanopatterning, nanocluster, nanostructure, nanometer-scale, titanium

15. NUMBER OF PAGES

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	

Parallel Fabrication and Electronic Characterization of Nanostructured and Nanoheterostructured Metal Surfaces

AFOSR Grant NO. F49620-96-1-0007

Final Report

March 31, 1999

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Original Objectives

- generation of selectively nanostructured substrates
- integration of nanostructures with silicon-based microelectronics
- electronic properties of metal nanostructures

Status of Effort

Among the more notable accomplishments during the course of this contract we identify the following results:

- **Formation of Ordered Nanocluster Arrays by Self-Assembly on Nanopatterned Si(100) Surfaces.**

A precisely ordered and precisely located array of 5 nm diameter nanoclusters has been fabricated by first etching into the substrate an array of holes with diameters comparable to the size of nanoclusters sought and then depositing adatoms on the substrate. The severely restricted diffusion field defined by the holes dominates nucleation and growth to produce a single nanocluster in each etched hole. Using Low Energy Electron Enhanced Etching (LE4) in a DC hydrogen plasma, we transferred an hexagonal array of 18 nm diameter holes with a 22 nm lattice constant from a biologically derived mask into Si(100). After etching, the mask was removed, and the patterned surface was intentionally oxidized in an oxygen plasma. Deposition of 1.2 nm of Ti on the oxidized surface produced an ordered array of 5 nm diameter metal nanoclusters positioned at the etched hole sites. Our methods achieved massively parallel processing at the key fabrication steps of pattern generation, pattern transfer, and nanocluster formation. Therefore, our methods enable rapid fabrication of arrays for fundamental studies and provide a route to manufacturability of nanostructure arrays for technological purposes.

- **Controlled Morphology of Biologically Derived Metal Nanopatterns**

We reported the ability to control the morphology of nanometer thick Ti oxide films which were created via a parallel nanofabrication process using a two-dimensional protein crystal as a template. Atomic force microscopy was used to examine the evolution of these structures from a periodic array of nanometer-scale dots (nanodots) to a screen containing a periodic array of nanometer-scale holes (nanoscreen) as the film thickness was increased. A Monte Carlo solid-on-solid simulation was then developed to explain the thickness dependence of the morphology as the metal film self-organizes into these nanopatterns.

- **Creation of Nanometer Scale Patterns with Selected Metal Films**

The goal of these experiments was three-fold: (1) to determine if some of the metals near Ti in the periodic table might have pattern forming properties similar to that of titanium, (2) to gain the ability to predict which additional metals might form nanodots and (3) to understand why some metals produce patterns, but not others. We reported the creation of large arrays of nanometer-scale dots (nanodot arrays) with several of these of these transition metals. The correlation between nanodot array formation and the interaction energies between metal atoms, other metals atoms and the surface of the sample was explored. The behavior of the metal films was then investigated with the aid of a Monte Carlo solid-on-solid simulation.

- **Numerical Simulation of the Evolution of Nanometer-Scale Surface Topography Generated by Ion Milling**

Using the theory of ripple topography proposed by Bradley and Harper, a computer simulation of nanoscale hole formation based on curvature dependent sputtering and surface self-diffusion was formulated. To determine the validity of this model as applied to the patterning process, profiles of actual nanoscale etch pits were obtained using atomic force microscopy, and the simulation has been used to evolve these profiles in time. Even with a number of simplifying assumptions in place, the model simulates the experimental data quite well.

Personnel Supported During this Contract:

Faculty:	Dr. Kenneth Douglas; Dr. John Price
Post-Docs:	Dr. Patrick Sullivan; Dr. Thomas A. Winningham
Graduate Students:	Thomas A. Winningham, awarded Ph.D. in May,1997; Jon T. Moore, awarded Ph.D. in December,1997
Other:	None

Publications

Accepted

"Formation of Ordered Nanocluster Arrays by Self Assembly on Nanopatterned Si(100) Surfaces" Thomas A. Winningham, H.P. Gillis, D.A. Choutov, Kevin P. Martin, Jon T. Moore, and Kenneth Douglas, Surf. Sci. **406**, 221 (1998).

"Creation of Nanometer Scale Patterns With Selected Metal Films," J. T. Moore, P. D. Beale, T. A. Winningham, K. Douglas, Appl. Phys. Lett., 72, 1840 (1998).

"A numerical simulation of the evolution of nanometer-scale surface topography generated by ion milling," Thomas A. Winningham, Noel A. Clark, and Kenneth Douglas, J. Vac. Sci.Technol. A **16**, 1178 (1998).

"Controlled Morphology of Biologically Derived Metal Nanopatterns, " Jon T. Moore, Paul D. Beale, Thomas A. Winningham, Kenneth Douglas, Applied Physics Letters **71**, 1264-1266 (1997).

"Fused-Salt Electrodeposition of Thin-Layer Silicon, " J.T. Moore, T.H. Wang, M.J. Heben, K. Douglas, T.F. Ciszek, Proceedings of the 26th IEEE Photovoltaic Specialists Conference, pp.775-778 (1997).

"Parallel Nanofabrication Using Microbial S-Layers," Kenneth Douglas, Noel A. Clark, Jon T. Moore, Thomas A. Winningham, Samuel Levy, Ivar Frithsen, Jacques Pankove, Paul Beale, Harry P. Gillis, D.A. Choutov, and Kevin P. Martin, FEMS Microbiology Rev.,(20) 1-2, p. 151-174 (1997).

"Biomimetic Approaches to Nanostructural Fabrication," K. Douglas, in Biomimetic Materials Chemistry, p. 117, edited by Stephen Mann, VCH Publishers, New York, (1996).

Interactions/Transitions

participation/presentations at meetings, conferences, seminars, etc.

"Ordered Nanocluster Arrays Grown in Confined Geometries," Materials Reserach Society Meeting, Boston, MA (January, 1998).

"Fused-Salt Electrodeposition of Thin-Layer Silicon," 26th IEEE Photovoltaic Specialists Conference, Anaheim,CA (September, 1997).

"Nanopattern Transfer Using Protein Crystal Masks: A Numerical Simulation of Experimental Results ", American Vacuum Society's 44th National Symposium, San Jose, CA (October, 1997)

Invited: "Patterned Protein Crystal/Metal Oxide Composite Films," Patterned Organic Thin Films Conference sponsored by American Chemical Society, Roanoke, VA, (October, 1997).

Invited: "Increasing Critical Current Density in High Temperature Superconductors by Creating Regular Structure of Flux Pinning Sites Using Biological Nanolithography," Flux, Quantum, and Mesoscopic Effects in Superconducting Materials and Devices Workshop sponsored by the AFOSR, Santa Fe, NM, (August, 1997).

"Ordered Nanocluster Array Formation on Si(100) Surfaces Patterned by Low Energy Electron Enhanced Etching through Biological Masks," Optical Society of America Topical Meeting on Chemistry and Physics of Small Structures, Santa Fe, NM, (February, 1997).

Invited: "Parallel Nanofabrication Using Microbial S-Layers," Fourth International Workshop on Microbial S-Layers, Rothenburg, Germany, (November, 1996).

consultative and advisory functions to other laboratoraties and agencies

None

transitions

None

New Discoveries, Inventions, or Patent Disclosures

"Method for Altering the Aperture Diameters in Nanoscale Patterning Masks (Nanomasks) for the Fabrication of Semiconductor Quantum Dots of Varying Sizes: Tunable Nanomasks", Thomas A Winningham, Kenneth Douglas, and Jacques Pankove, invention disclosure filed (1998).

"Silicon Quantum Dot Laser", K. Douglas, J. Pankove, and G. Moddel, U.S. Patent #5,703,896 (1997).

"Silicon Quantum Dot Laser", K. Douglas, J. Pankove, and G. Moddel, U.S. Patent #5,559,822 September 24, 1996.

Honors/Awards

Faculty Sponsor:

American Western University Fellowship

Jon Moore September 1996 - May 1998